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SILICON AZIDE FIREPROOF MATERIAL

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DETAILED EXPLANATION OF THE INVENTION

This invention pertains to silicone azide fireproof material made from already-produced silicone azide, to which finely-powdered material which contains analytically 1.5% or more by weight of titanium oxide as the sintering agent is added, in such a manner that the titanium oxide content of the product is analytically 0.2 - 25%.

*Numbers in margin indicate pagination in foreign text.
Since silicone azide itself does not have the sintering property, finely pulverized silicone is used as the forming agent in order to produce a silicone azide fireproof material, which is then nitrided and sintered in the ordinary way.

With this method, it is not possible to make large fireproof material with precise detailed because finely powdered silicone is used and the entire formed body has to be nitrided and sintered. Furthermore, when fine silicone powder is added to silicone azide to carry out nitriding and sintering, adding clay for plasticity makes the product easily breakable during sintering, in general.

This invention attempts to overcome these shortcomings, whereby already-prepared silicone azide is mixed with finely-powdered raw material which analytically contains 1.5% or more by weight of titanium oxide, so that the final titanium oxide content would be analytically 0.2 - 25% by weight. This combined material is formed into the desired shape for sintering; it could also be stamped into templates of desired shapes at the place where the silicone azide fireproof material is actually used.

According to this invention, this sintering property could be obtained for sintering in either an oxidizing or a nitriding atmosphere. Furthermore, it is advantageous because when titanium oxide and clay are combined, no tendency towards breakage was noted.

In carrying out this invention, the unique properties of silicone azide tend to be lost if more than 25% by weight of titanium oxide is added; thus there is no advantage. However, when titanium oxide is added singularly, the final titanium oxide content should be over 4% by weight if good sintering is to be expected, but if titanium oxide coexists with iron oxide, calcium oxide, sodium oxide, potassium oxide, etc., good sintering results can be obtained even if the final titanium oxide concentration is below 4% by weight. With titanium oxide contents below 0.2% by weight, good sintering is possible if iron oxides or oxides of alkaline or alkaline-earth metals coexist; but if such oxides are not present, and other oxides such as silicone oxide or aluminum oxide coexist, the sintering property lessens rapidly, and closely-organized silicone azide fireproof material could not be formed. Accordingly, it is desirable to hold the titanium oxide contents in the
The final product to between 0.2 - 25% by weight.

If the titanium oxide content in the finely-powdered material added to silicone azide is reduced to below 1.0% by weight, virtually no effect as a sintering material is recognizable. At least 1.5% by weight or more is necessary.

These finely pulverized materials could be of one kind or be more than one. If more than one, they can be used separately or in combination, and addition could be done simultaneously or separately. From the standpoint of the sintering property, it is desirable that particular sizes of these finely-pulverized materials which contain 1.5% by weight or more of titanium oxide be as small as possible. At any rate, 50% by weight or more should be ultramicro particles below 100 μ.

When the form desired is simple, or the final product is small in size, the additives mentioned above would suffice. But when large-sized or complex-shaped products are formed, inorganic bonding material such as clay phosphate, silica gels and sols such as snowtex, or organic bonding material such as CMC, lignin sulfonate salts, and various resins such as used in ordinary fireproof material could be added in order to improve plasticity and strength when dried.

The fireproof material made by this invention retains the chemical and physical characteristics of silicone azide, and in addition possesses an excellent bonding capability, so that it is useful for making melting furnaces of various metals, refining furnaces, and for the formation of steel masses.

Examples of practical uses of this invention are shown below, but this is meant to be an illustration, and no restrictions by any means are intended.
Table 1: Mixing proportions of examples (% by weight)

<table>
<thead>
<tr>
<th>Examples of practical use</th>
<th>Silicone azide</th>
<th>Titanium oxide</th>
<th>Titanium silicate</th>
<th>Silimanite</th>
<th>Wood Grain Clay</th>
<th>Silicone</th>
<th>Atmosphere for sintering</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3-1 mm and below</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>50</td>
<td>40</td>
<td>10</td>
<td></td>
<td></td>
<td></td>
<td>oxidizing</td>
</tr>
<tr>
<td>2</td>
<td>40</td>
<td>45</td>
<td>15</td>
<td>15</td>
<td>10</td>
<td>15</td>
<td>oxidizing</td>
</tr>
<tr>
<td>3</td>
<td>50</td>
<td>25</td>
<td>15</td>
<td>10</td>
<td>5</td>
<td></td>
<td>nitrizing</td>
</tr>
<tr>
<td>4</td>
<td>45</td>
<td>40</td>
<td>10</td>
<td></td>
<td></td>
<td>15</td>
<td>nitrizing</td>
</tr>
<tr>
<td>comparison specimen</td>
<td>45</td>
<td>40</td>
<td></td>
<td>15</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 2 shows some physical properties of fireproof material made from the examples presented above. Their properties are appreciably better than those of the comparison material. The sintering temperature is 1400°C and 1450°C for oxidizing and nitrizing, sintering, respectively.

Table 2: Physical Properties of Fireproof Materials

<table>
<thead>
<tr>
<th>Examples of practical use</th>
<th>Porosity rate, %</th>
<th>Comparative weight per mass</th>
<th>Strength against Pressure</th>
<th>Sporing resistance (1200 water cooling)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>23.6</td>
<td>2.30</td>
<td>800</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>25.0</td>
<td>2.33</td>
<td>1300</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>27.2</td>
<td>2.31</td>
<td>1000</td>
<td>no peeling off up to 10 trials</td>
</tr>
<tr>
<td>4</td>
<td>28.9</td>
<td>2.27</td>
<td>850</td>
<td></td>
</tr>
<tr>
<td>comparison specimen</td>
<td>31.7</td>
<td>2.22</td>
<td>750</td>
<td></td>
</tr>
</tbody>
</table>

The chemical compositions of materials used are shown in Table 3.
<table>
<thead>
<tr>
<th>Material</th>
<th>Ignition Loss</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>TiO₂</th>
<th>Fe₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>Na₂O</th>
<th>K₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>silicone azide</td>
<td></td>
<td>Si 56.2</td>
<td>Al 2.9</td>
<td>Fe 2.3</td>
<td>N 36.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>silicone</td>
<td>Si 98.0</td>
<td>Al 0.1</td>
<td>Fe 1.0</td>
<td>Ca 0.2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>titanium oxide</td>
<td></td>
<td></td>
<td>over 98</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>titanium slug</td>
<td></td>
<td>8.6</td>
<td>6.3</td>
<td>65.1</td>
<td>12.0</td>
<td>1.0</td>
<td>5.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>sillimanite</td>
<td>2.4</td>
<td>15.8</td>
<td>76.0</td>
<td>3.3</td>
<td>1.1</td>
<td>-</td>
<td>0.3</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>wood grain clay</td>
<td></td>
<td>14.9</td>
<td>47.7</td>
<td>33.9</td>
<td>1.7</td>
<td>1.6</td>
<td>0.3</td>
<td>0.2</td>
<td>0.2</td>
</tr>
</tbody>
</table>